MTL TR 89-31

AD

THE RECRYSTALLIZATION AND RESPHEROIDIZATION OF TUNGSTEN GRAINS IN A TUNGSTEN-HEAVY ALLOY

AD-A207 647

ROBERT J. DOWDING
MATERIALS PRODUCIBILITY BRANCH

April 1989

Approved for public release; distribution unlimited.

US ARMY
LABORATORY COMMAND
MATERIALS TECHNOLOGY LABORATORY

U.S. ARMY MATERIALS TECHNOLOGY LABORATORY
Watertown, Massachusetts 02172-0001

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (*When Data Entered*)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER MTL TR 89-31	2. GOVT ACCESSION NO	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (*and Subtitle*) THE RECRYSTALLIZATION AND RESPHEROIDIZATION OF TUNGSTEN GRAINS IN A TUNGSTEN-HEAVY ALLOY		5. TYPE OF REPORT & PERIOD COVERED Final Report 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Robert J. Dowding		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001 SLCMT-MEM		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS D/A Project P612105.H84
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Laboratory Command 2800 Powder Mill Road Adelphi, Maryland 20783-1145		12. REPORT DATE April 1989 13. NUMBER OF PAGES 17
14. MONITORING AGENCY NAME & ADDRESS (*if different from Controlling Office*)		15. SECURITY CLASS. (*of this report*) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (*of this Report*)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (*of the abstract entered in Block 20, if different from Report*)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (*Continue on reverse side if necessary and identify by block number*)

Tungsten alloys
Recrystallization
Heavy metals
Grain structures
Annealing

20. ABSTRACT (*Continue on reverse side if necessary and identify by block number*)

(SEE REVERSE SIDE)

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (*When Data Entered*)

Block No. 20

ABSTRACT

Refinement of the microstructure of tungsten-heavy alloys, specifically 90 W-7 Ni-3 Fe, has been achieved through heavy cold working followed by a heat treatment that recrystallized and respheroidized the tungsten grains. Hydrostatic extrusion of the alloy to reductions of area of 83% and 96% followed by isothermal annealing gives fine, spherical tungsten grains imbedded within the nickel-iron matrix. The finest grain size achieved was 15 microns for the 96% extrusion when subjected to an anneal at 1300°C for 2 hours. The apparent activation energy for the process was found to be 530 kJ/mole. This value indicates that volume diffusion is significant in respheroidizing the grains.

CONTENTS

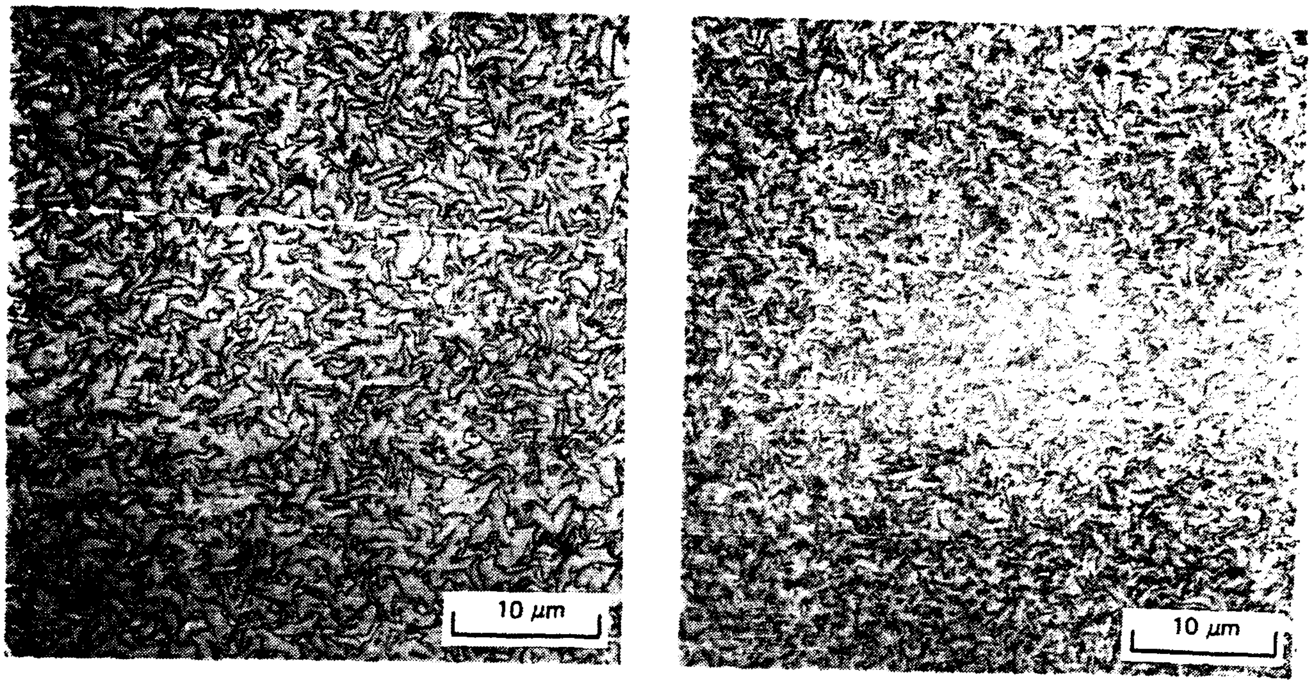
INTRODUCTION

Tungsten-heavy alloys are two-phase mixtures, genuine composites, of tungsten grains in a matrix of nickel and iron, supersaturated with tungsten. These alloys are produced by the blending of elemental powders followed by liquid phase sintering. Upon sintering the resulting tungsten grain diameters are traditionally in the range of 30 to 50 microns.[1] The grains are nearly pure containing only 0.3% Ni-Fe while the matrix contains about 26% tungsten with the balance being nickel and iron in the original proportion.[2] These large grain diameters occur despite the use of tungsten powder with initial particle sizes of less than 5 microns. The tungsten grain growth takes place, during sintering, in the presence of the liquid phase by a number of mechanisms collectively referred to as Ostwald ripening.[3,4] Tungsten at high-energy sites (small grains and sharp corners) is dissolved into the nickel-iron matrix, diffused, and reprecipitated at low energy locations, typically larger tungsten grains. This leads to the elimination of the smaller grains, growth or ripening of the larger ones, and an overall reduction in the total energy of the system.[3,5]

The strength dependence of a single-phase metallic substance upon grain size is well known. It has been demonstrated (e.g., Hall-Petch) that finer grain sizes result in higher strengths.[6-8] The grain size of any metal may be refined by cold working followed by recrystallization.[9] In most cases, this requires that the cold-worked structure be given a high temperature treatment for a specified length of time. The resulting grain size will be determined by several factors. They include the degree of cold work, the temperature of recrystallization, and the time at temperature, among others.[6,7,10] It may be possible to strengthen this two-phase tungsten alloy by grain refinement. It will be shown that it is possible to achieve the fine, spherical, tungsten grains that would likely be necessary.

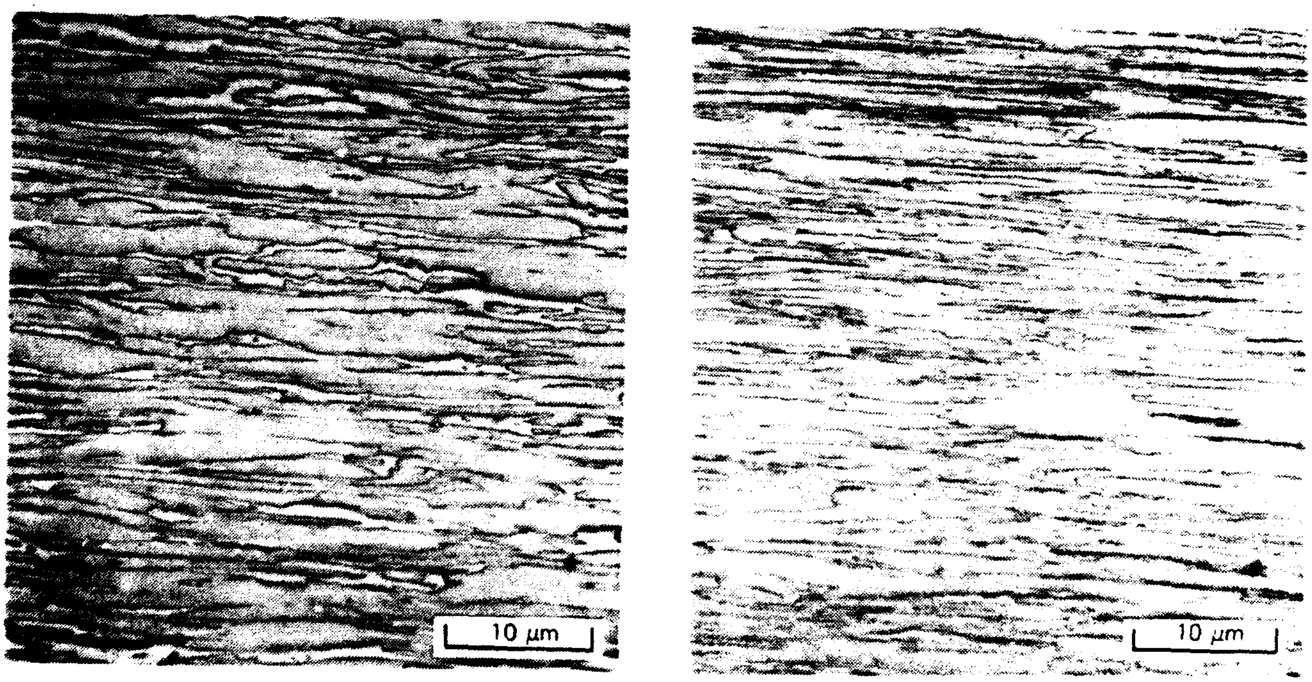
The method for refining the spherical grain size is cold working followed by a heat treatment that leads to recrystallization. This process should produce fine, spherical grains of tungsten in this alloy. In the hydrostatically extruded material studied here, the size of these grains can be estimated if the original grain size and extrusion ratio are known and volume conservation is assumed. There are other considerations regarding the nature of the extruded grain and its structure after heat treatment. The premise is that the extruded grain is recrystallized into a bamboo-like structure and the bamboo cells are the precursors to the refined spherical grains. Using area and volume relations for these two shapes and the previous assumptions, 50-micron diameter grains will become 20-micron and 10-micron diameter grains for 6:1 and 24:1 extrusions, respectively. Figures 1 and 2 illustrate the irregular shape of the extruded grains that is not accounted for in this calculation.

1. GERMAN, R. M., HANAFEE, J. E., and DiGIALLONARDO, S. L. *Toughness Variation with Cooling Rate for Liquid Phase Sintered W-3.5 Ni-1.5 Fe.* Met. Trans. A., v. 15A, January 1984, p. 121.
2. GURWELL, W. E., NELSON, R. G., DUDDER, G. B., and DAVIS, N. C. *Fabrication and Properties of Tungsten Heavy Alloys Containing 30% to 90% Tungsten.* Battelle Pacific Northwest Laboratory Report PNL-5218, September 1984.
3. KAYSSER, W. A., and PETZOW, G. *Present State of Liquid Phase Sintering.* Powder Metallurgy, v. 28, no. 3, 1985
4. YOON, D. N., and HUPPMANN, W. J. *Chemically Driven Growth of Tungsten Grains During Sintering in Liquid Nickel.* Acta Met., v. 27, 1979, p. 973.
5. LENEL, F. V. *Powder Metallurgy, Principles and Applications.* MPIF, 1980.
6. SHEWMON, P. G. *Transformations in Metals.* McGraw-Hill Book Company, New York, 1969.
7. DIETER, G. E. *Mechanical Metallurgy.* McGraw-Hill Book Company, New York, 1979.
8. FLEMINGS, M. C. *Solidification Processing.* McGraw-Hill Book Company, New York, 1974.
9. CARTER, G. F. *Principles of Physical and Chemical Metallurgy.* American Society for Metals, 1979.
10. BARRETT, C. R., NIX, W. D., and TETLEMAN, A. S. *The Principles of Engineering Materials.* Prentice-Hall, Inc., New Jersey, 1973.



6:1 As Received, As Polished, Mag. 200X

Figure 1. Transverse section of extruded 90 W alloy.



24:1 As Received, As Polished, Mag. 200X

Figure 2. Longitudinal section of extruded 90 W alloy.

The samples used in this study had a nominal composition of 90% tungsten, 7% nickel, and 3% iron and shall be referred to as 90 W. These samples were hydrostatically extruded at ratios of 6:1 and 24:1 (approximately 83% and 96% reduction of area, respectively). The need for determining the various conditions that form the new grains lies in the fact that previous recrystallization studies did not investigate this type of cold work or these levels of reduction.[11-13] Dieter[7] states that recrystallization behavior is influenced by the amount of prior deformation and "for a given reduction in cross section, different metalworking processes ...produce different effective deformation." As a result, different recrystallization behavior may be observed than was for previous studies, affecting the final grain size.

Up to this point, the terms recrystallization and respheroidization have been used as synonyms. The difference needs to be emphasized as it will become apparent that they indicate different processes here. Recrystallization, in broad terms, is the formation of new, strain-free grains from cold-worked grains. This usually results in grain refinement.[7] Respheroidization shall, for the purposes of this report, refer to the formation of new, round, tungsten grains within the matrix. The spherical grains are derived from the recrystallized grains and result in the lowest surface energy condition for the tungsten in the alloy.

There have been prior efforts to identify the recrystallization of worked tungsten grains in heavy alloys as summarized in Table 1. From these previous investigations, it is seen that the recrystallization temperature of the tungsten in the heavy alloy is dependent upon the amount of cold work and the alloy content.

Table 1.

Alloy	Cold Work (%)	Method	Rextal Temp. (°C)	Method of Identification	Ref.
90 W-7 Ni-3 Fe	2 - 50	Upset	1100 - 900	Microhardness	12
95 W-3.5 Ni-1.5 Fe	25	Rolled	1200	Unknown	13
90 W-7 Ni-3 Fe	5 - 90	Rolled	1000 - 800	Tensile Test	11

EXPERIMENTAL PROCEDURE

The samples of hydrostatically-extruded 90 W tungsten-heavy alloy were supplied in the as-extruded condition. These samples had received 6:1 and 24:1 reductions in cross-sectional area and the final diameter of the specimens was 0.125" and 0.065", respectively. The samples were given isothermal heat treatments in a 2-inch (51 mm) diameter tube furnace with a flowing dry hydrogen atmosphere (dew point -25°C). The annealing temperatures chosen were 1300°C, 1350°C, 1400°C, and 1450°C. The temperature was controlled to within 5°C. The times at temperature were 1/2, 1, 2, and 4 hours. The liquidus temperature for the matrix composition of this alloy lies between 1400°C and 1450°C, likely being very close to the latter.[14] All of the samples were prepared for optical metallography using standard techniques. The unetched microstructures were photographed and the apparent grain sizes and shapes were evaluated by calculating the tungsten grain surface area to volume ratio.[15]

11. YODOGAWA, M. *Effects of Cold Rolling and Annealing on the Mechanical Properties of 90 W-7 Ni-3 Fe Heavy Alloys.* Sintering - Theory and Practice, D. Kolar, S. Pejovnik, and M. M. Restic, ed., Elsevier Scientific Publishing Co., 1982.
12. FRANTSEVICH, I. N., TEDOROVICH, O. K., and BAZHENOVA, L. G. *Recrystallization of Tungsten in Tungsten-Nickel-Iron Alloys, Part I.* Soviet Journal of Powder Metallurgy, Metal Ceramics, v. 6, 1967, p. 393.
13. NORTHCUTT, W. G., JOHNSON, D. H., FERGUSON, J. E., and SNYDER, W. B. *Variables Affecting the Properties of Tungsten-Nickel-Iron Alloys.* ARPA/ARCOM Kinetic Energy Ammunition Materials Panel and U.S. Army Materials and Mechanics Research Center (AMMRC) Conference, Charlottesville, VA, May 24-27, 1976.
14. WINSLOW, F. R. *The Nickel-Iron-Tungsten Phase Diagram.* Y-12 Plant Report No. 1785, Oak Ridge, Tennessee, 1971.
15. Metals Handbook, 9th ed., v. 9, 1985.

This was done using a line intercept method in the directions parallel and perpendicular to the extrusion direction.

RESULTS AND DISCUSSION

Recrystallization of the extruded tungsten grains takes place at a temperature much lower than those investigated here. Frantsevich, et al.,[12] reports that in 90 W-7 Ni-3 Fe, which had been cold worked by upsetting 2% to 50%, the tungsten grains recrystallize at 1100°C to 900°C, respectively. Working with 95 W-3.5 Ni-1.5 Fe, cold rolled 25%, Northcutt, et al.,[13] observed the recrystallization of the tungsten at 1200°C. Further, Yodogawa[11] shows significant loss of tensile strength in the temperature range 1000°C to 800°C for 90 W-7 Ni-3 Fe that had been cold rolled 5 to 90 percent. Since the recrystallization temperature is dependent upon the amount of prior cold work,[7] it is expected that the tungsten in these hydrostatically extruded samples recrystallized at or below 900°C. This is especially true of the samples that received the 24:1 reduction, because none of the previous studies examined samples that had 96% reduction of area.

Recrystallization of the tungsten grains was confirmed by X-ray diffraction. The sample chosen had been extruded to a reduction of 6:1 and had been given the shortest time and temperature treatment and, therefore, had the highest probability of showing the prior cold-worked structure. Measured at the half height, broad diffraction peaks observed from (310) planes in the as-extruded sample, were seen to become more narrow in the annealed sample. Similar behavior was seen for (110) and (200) planes. This is an indication that recrystallization had taken place.

Additionally, three pieces of the 6:1 extrusion were annealed for 1 hour at 900°C, 1000°C, and 1100°C. Figures 3a through 3c are photomicrographs of the resulting structure. It can be seen at 1000°C that at least partial recrystallization has taken place. Also, at 1100°C it is seen that the tungsten is fully recrystallized and that a bamboo-like grain structure has formed.

In the process of respheroidization, the first step is a growth of the recrystallized grains in a direction lateral to the extrusion direction; this is also seen at 1000°C (Figure 3b). Growth of the tungsten in this manner was also observed by Pugh[16] in his studies of heavily drawn pure tungsten. Pugh[17] also stated that the elongated grains of pure tungsten contained a partially or completely polygonized sub-structure prior to heat treatment which contributed to the ease of formation of the subgrains at slightly elevated heat treating temperatures. Further, it is postulated by Pugh that polygonization occurs at approximately 600°C. The second step in respheroidization is the formation of the "bamboo" structure as seen at 1000°C in Figure 3b. The bamboo cells are single crystal grains of tungsten with high angle grain boundaries with their neighbors. Lastly, these boundaries are penetrated by the matrix to form discrete tungsten grains within the matrix, which is seen at the elevated temperatures studied here.

16. PUGH, J. W. *On the Recovery and Recrystallization of Tungsten.* Proceedings of the 3rd Plansee Seminar, F. Benesovsky, Reutte/Tirol, Ju., 1958.
17. PUGH, J. W. *The Temperature Dependence of Preferred Orientation in Rolled Tungsten.* Trans. Met. Soc. of AIME, v. 212, 1958, p. 637.

(a) One Hour at 900°, Mag. 550X

(b) One Hour at 1000°, Mag. 550X

(c) One Hour at 1100°. Mag. 550X

Figure 3. 6:1 extruded 90 W alloy after annealing.

The kinetics for the respheroidization and growth of the tungsten grains was observed to vary with both time at temperature and the extrusion ratio (% cold work). For the 6:1 extrusions, the onset of respheroidization was detected at 1300°C after 2 hours and similarly after a 1/2 hour at 1350°C. These are the times and temperatures at which the tungsten grains take on a spherical shape. This can be seen in Figure 4. The appearance of a uniform distribution of equiaxed grains occurs after 4 hours at 1300°C or 2 hours at 1350°C. It appears, from a visual inspection of the 1400°C and 1450°C series, after times as short as a 1/2 hour, the tungsten spheroids are in the growth stage already.

The 24:1 extrusions (Figure 5), show similar characteristics but the times and temperatures are shifted to lower values. This would be expected since there was a greater amount of cold working done to these samples. Onset of respheroidization occurs at 1300°C for 1 hour and this corresponds to the 1300°C 2-hour treatment for the 6:1 extrusion. For all other conditions of higher temperature and longer time, the 24:1 samples are in the equiaxed growth stage. The finest grain size observed was 15 microns for the 24:1 sample given a 1300°C 2-hour treatment.

When considering the respheroidization phenomenon of the 90 W alloy, the measurement of the grain size as a function of time and temperature was necessary. It was essential to identify the point where the grain growth stage began. Since the measurement of the grain size was particularly difficult to quantify, the critical points were identified by measuring the grain surface area to grain volume ratio (S/V). This measurement was made in orthogonal directions, parallel and transverse to the extrusion direction. For the elongated grains and any nonspherical grain shape this was only an "effective" ratio. The advantage to this measurement was that the grain shape (aspect ratio) as well as size could be defined at once. A high value of S/V implied a small effective grain size and conversely a low value of S/V meant a large effective grain size. The point at which the two perpendicular S/V ratios became equal was defined as the conditions where equiaxed grains developed. Figures 6 and 7 graphically show the change in the S/V ratio with time for each heat-treating condition and extrusion ratio. The error in the S/V ratio is expected to be less than ±0.021/mm and ±0.028/mm for the 6:1 and 24:1 extrusions, respectively. The possibility of measurement error was considered when determining the occurrence of equiaxed grains. The points where equiaxed grains first appear are summarized in Table 2. The existence of equiaxed grains at different temperatures or times due to this amount of cold work is not unusual or unexpected.[6]

Table 2

Temperature (°C)	6:1 (hr)	24:1 (hr)
1300	4	2
1350	2	1
1400	< 1/2	< 1/2
1450	< 1/2	< 1/2

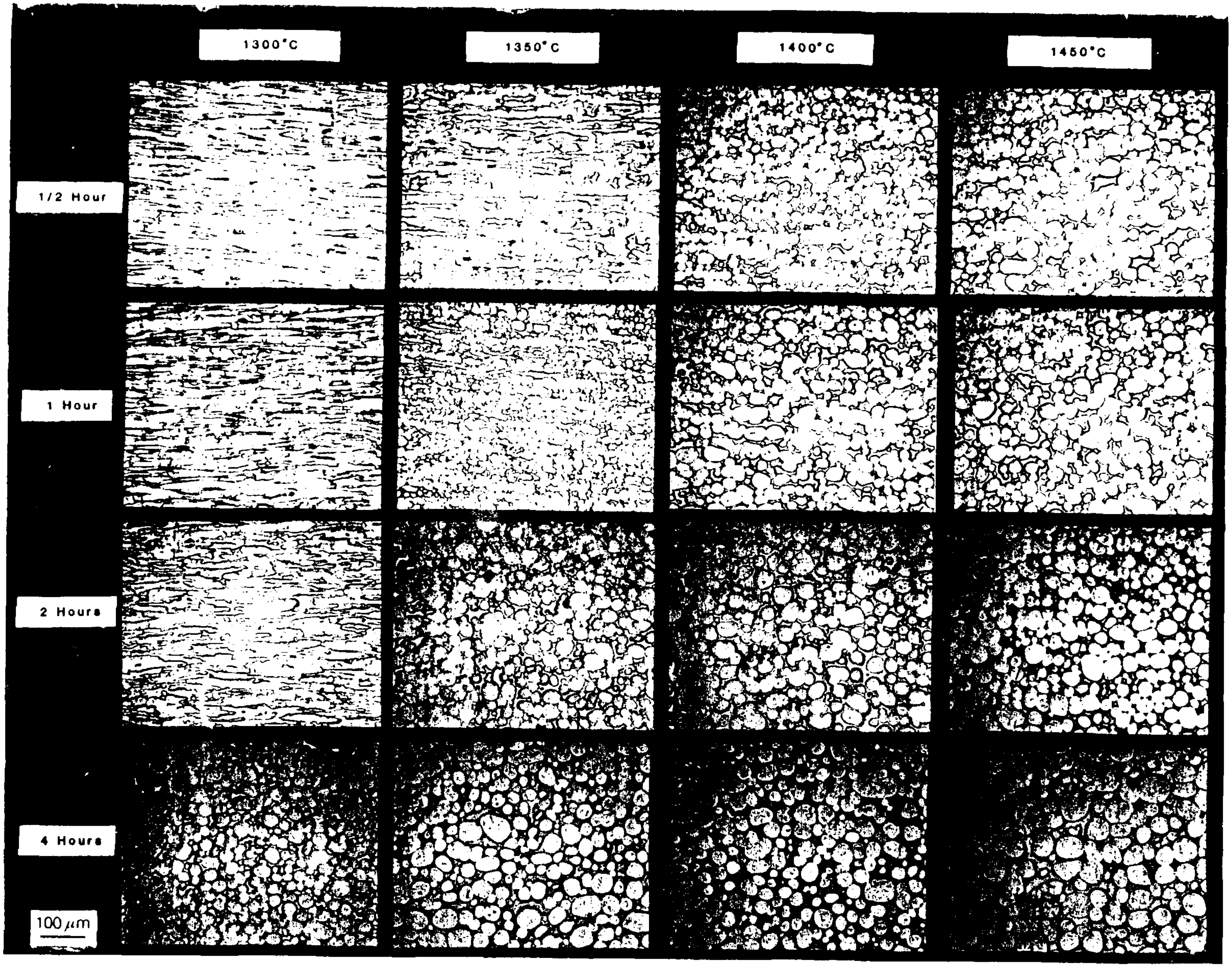


Figure 4 Effect of time and temperature on the recrystallization of extruded 90 W alloy. Extrusion ratio 6:1.

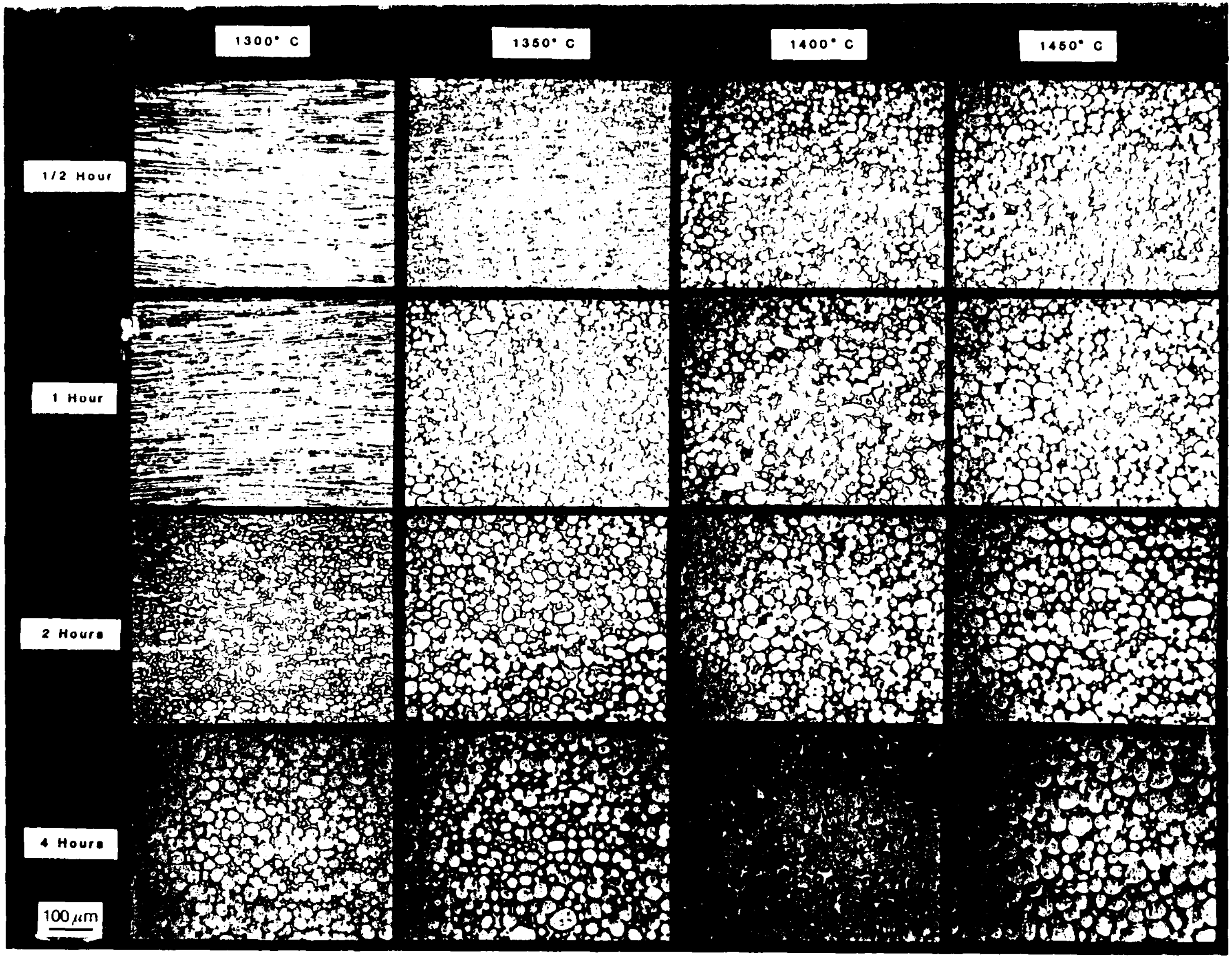


Figure 5. Effect of time and temperature on the recrystallization of extruded 90 W alloy. Extrusion ratio 24:1

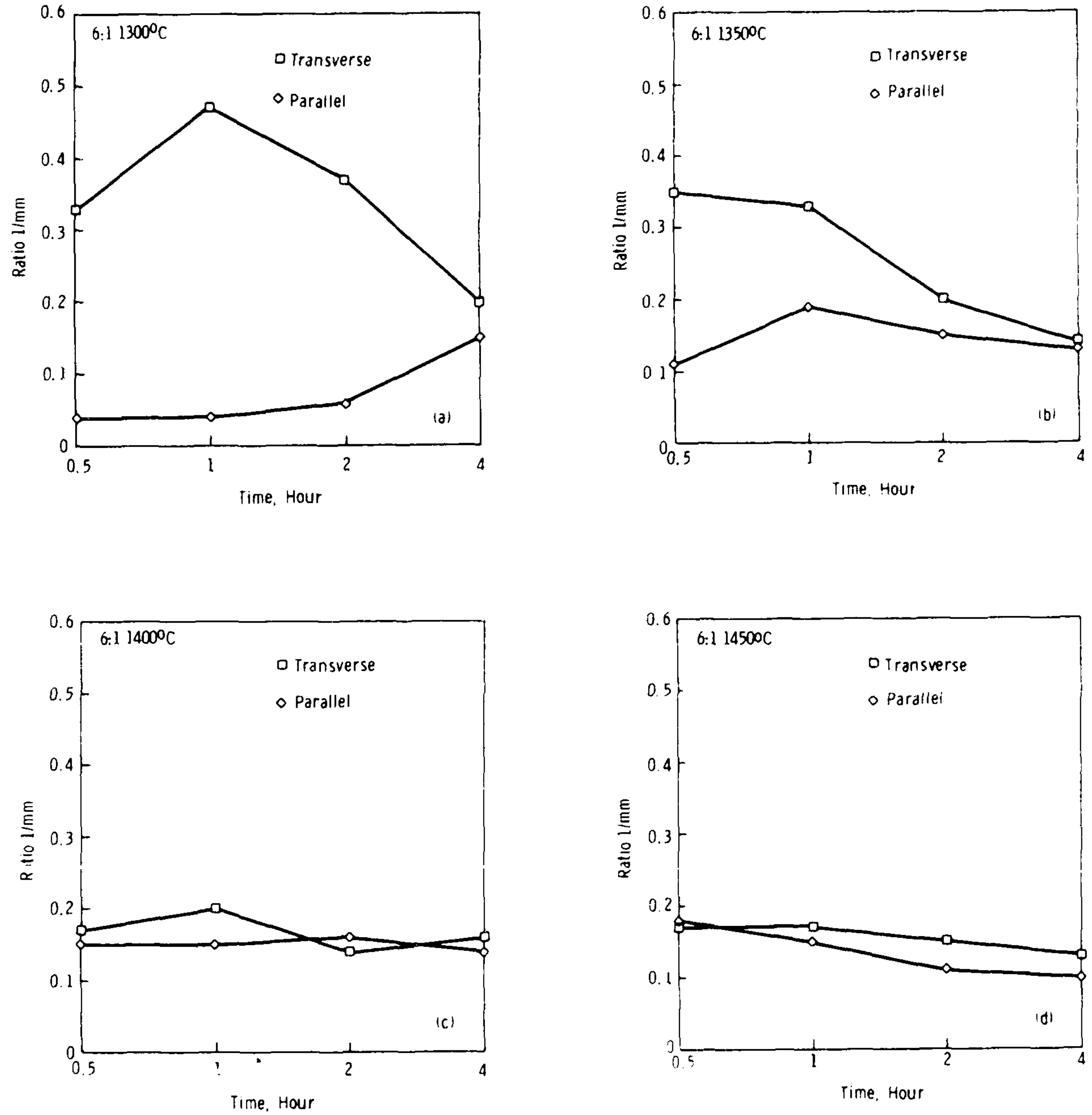


Figure 6 The change in the tungsten grain surface-to-volume (S/V) ratio in the longitudinal and transverse directions with time Extrusion ratio 6:1

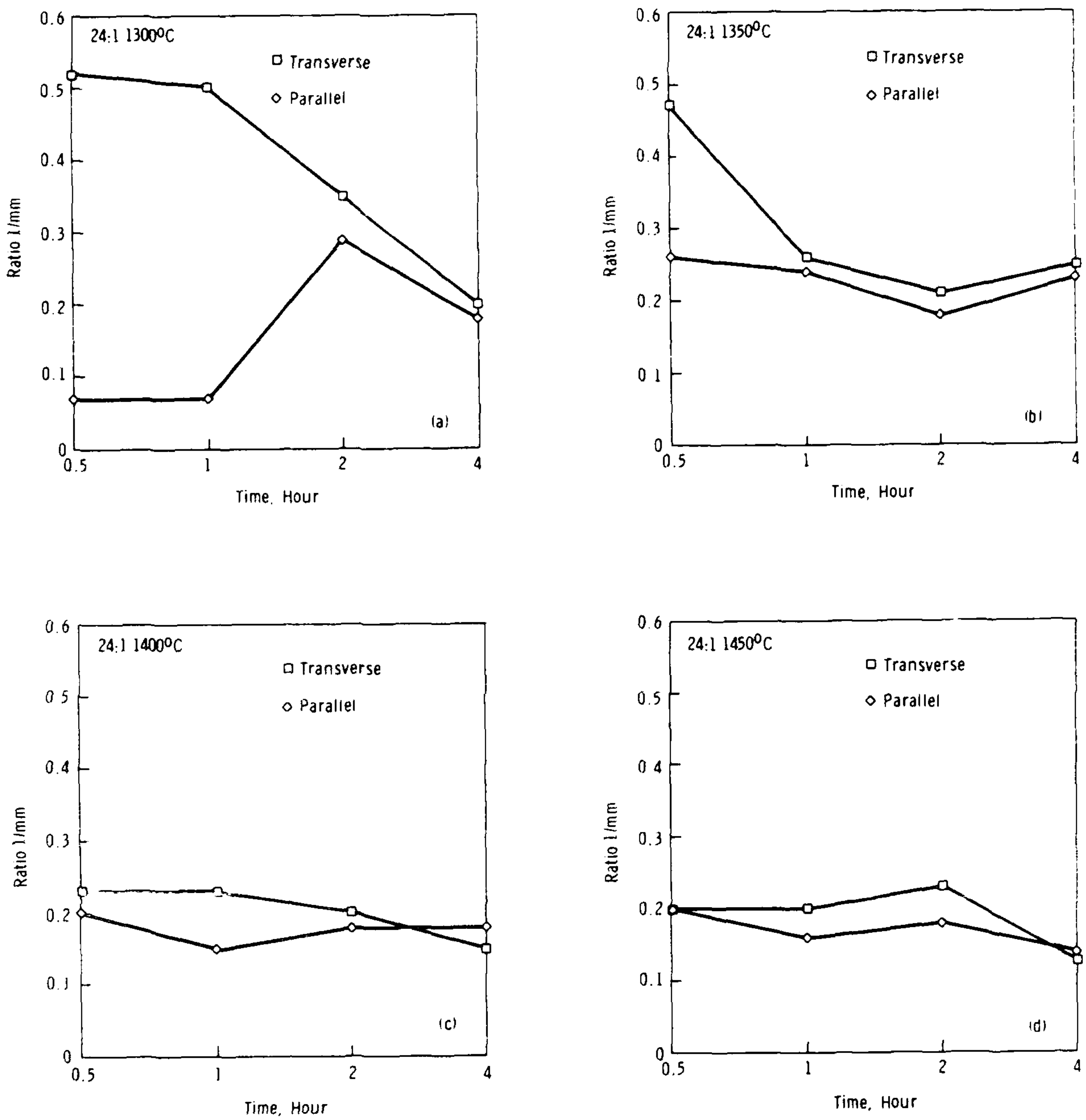


Figure 7 The change in the tungsten grain surface-to-volume (S/V) ratio in the longitudinal and transverse directions with time. Extrusion ratio 24:1.

The energy of deformation that is stored within the worked structure can be found by using high temperature stress-strain data for tungsten and calculating the total energy of deformation,[18,19] and by assuming that approximately 5% of that energy is retained.[20,21] An assumption of 5% is a conservative estimate. The retained energy is stored in the elastic stress fields around dislocations and point defects that were formed during deformation. This energy is released during high temperature annealing.[21] This calculation reveals that the stored energy will provide an equivalent temperature contribution of approximately 75°C. The implication is that a liquid phase structure could be seen in heavily cold worked and annealed tungsten-heavy alloy at temperatures 75 degrees below the liquid phase temperature. The liquid phase temperature of this alloy is very close to 1450°C. The structures seen at lower temperatures are a result of the stored energy of cold working.

For respheroidization or any thermally activated process, the apparent activation energy for that process can be calculated. The data used for these calculations comes from the S/V measurements (Appendix 1) which were used to draw the curves of Figure 8. Equation 1, adapted from the Arrhenius equation, describes the general form of part of those curves:

$$(S/V) = C + A \exp [Q/RT] \tag{1}$$

where C and A are constants, Q is the apparent activation energy that is being calculated, R is the universal gas constant (8.314 Joules/mole K) and T is the absolute temperature.

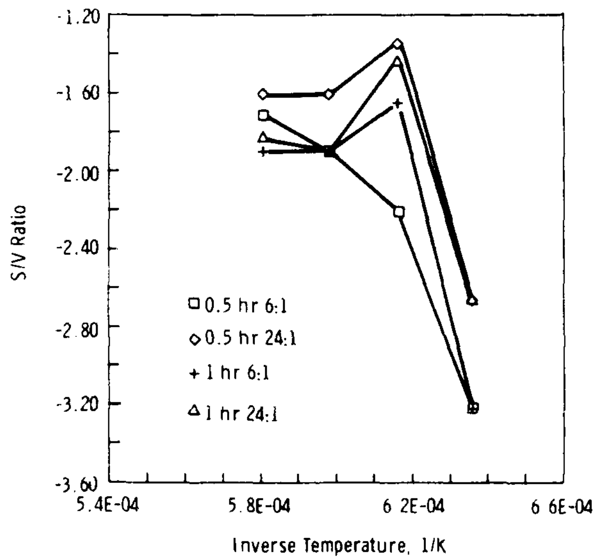


Figure 8. Arrhenius plot of Ln(S/V) versus reciprocal temperature.

18. KING, G. W., and SELL, H. G. *The Effect of Thoria on the Elevated-Temperature Tensile Properties of Recrystallized High-Purity Tungsten.* Trans. Met. Soc. of AIME, v. 233, 1965, p. 1104.
19. ZUKAS, E. G., and EASH, D. T. *Possible Reinforcement of the Tungsten-Nickel-Iron Composite with Tungsten Fibers.* J. Less Common Metals, v. 32, 1973, p. 345.
20. BEVER, M. B., HOLT, D. L., and TICHNER, A. L. *Progress in Materials Science.* Pergamon Press, Ltd., London, v. 17, 1973.
21. HONEYCOMBE, R. W. K. *The Plastic Deformation of Metals.* 2nd ed., American Society for Metals, 1984.

To calculate the activation energy for the spheroidization event, the natural logarithm of S/V was plotted versus the inverse of the absolute temperature (Figure 8). The measurements of S/V in the direction parallel to the extrusion direction were used because larger changes in structure were measured. In Figure 8, it is seen that there are two modes or stages occurring. The first is the formation of the new tungsten spheroids which takes place between 1300°C and 1350°C. The second is the growth of the grains which can be seen as the dip in the curve at those elevated temperatures. For each of these curves, an activation energy can be determined. The apparent activation energy for respheroidization was calculated to be 530 kJ/mole. German and Munir[22] indicate that for activated sintering of tungsten, a solid state process, the activation energy for densification was between 430 and 450 kJ/mole. That value was in agreement with previously published activation energies for grain boundary diffusion, 385 to 464 kJ/mole.[23-25] The respheroidization activation energy calculated here is higher than those values. This indicates a significant contribution of volume diffusion whose activation energy has been reported to be 628 kJ/mole.[26]

CONCLUSIONS

1. The respheroidization of 90 W-7 Ni-3 Fe, 6:1 extrusions, defined as the presence of equiaxed grains, occurs at 1300°C after 4 hours, at 1350°C after 2 hours, and at higher temperatures in less than a 1/2 hour.

2. The respheroidization of the same alloy, extruded 24:1, occurs after 2 hours at 1300°C and after 1 hour at 1350°C. At higher temperatures, the event is complete in less than a 1/2 hour.

3. The grain size of the tungsten grains in this heavy alloy can be refined by heavy cold working followed by an annealing treatment. The finest grain size achieved occurred for the 24:1 extrusion that had received a 1300°C 2-hour heat treatment. The grain size was 15 microns. It is conjectured that heat treatments at lower temperatures for longer times will lead to still finer grains, within the limits of the extruded grain diameters.

4. The activation energy for the respheroidization of the tungsten grains in this heavy alloy that was heavily cold worked, was found to be 530 kJ/mole. This value is higher than activation energy values for grain boundary diffusion indicating that volume diffusion is an important factor in the respheroidization.

ACKNOWLEDGMENTS

The author wishes to acknowledge the contributions of the following people: Mr. Richard Colena and Mr. Thomas Moynihan for assistance with the heat treatment; Ms. Carolyn Jones for the metallographic preparation of the samples; Dr. David Chipman for generating the X-ray diffraction data; Dr. Ralph P.I. Adler and Dr. Alan Goldman for valuable assistance in preparing the manuscript; and Mr. Stanley Lopata and Dr. Kenneth Tauer for technical guidance and discussion. The Samples of the hydrostatically extruded alloy were supplied by Mr. Robert Fiorentino of Battelle Memorial Institute, Columbus, Ohio.

22. GERMAN, R. M., and MUNIR, Z. A. *Enhanced Low Temperature Sintering of Tungsten.* Met. Trans. A., v. 7A, December 1976, p. 1873.
23. KOTHARI, N. C. *Sintering Kinetics in Tungsten Powder.* J. Less Common Metals, v. 5, 1963, p. 140.
24. VASILOS, T., and SMITH, J. T. *Diffusion Mechanism for Sintering Kinetics.* J. App. Phys., v. 35, no. 1, January 1964.
25. KREIDER, K. G., and BRUGGEMAN, G. *Grain Boundary Diffusion in Tungsten.* Trans. Met. Soc. of AIME, v. 239, 1967, p. 1222.
26. GREEN, W. V. *Short Time Creep-Rupture Behavior of Tungsten at 2250°C to 2800°C.* Trans. Met. Soc. of AIME, v. 215, 1959, p. 1057.

REFERENCES

1. GERMAN, R. M., HANAFEE, J. E., and DiGIALLONARDO, S. L. *Toughness Variation with Cooling Rate for Liquid Phase Sintered W-3.5 Ni-1.5 Fe.* Met. Trans. A., v. 15A, January 1984, p. 121.
2. GURWELL, W. E., NELSON, R. G., DUDDER, G. B., and DAVIS, N. C. *Fabrication and Properties of Tungsten Heavy Alloys Containing 30% to 90% Tungsten.* Battelle Pacific Northwest Laboratory Report PNL-5218, September 1984.
3. KAYSSER, W. A., and PETZOW, G. *Present State of Liquid Phase Sintering.* Powder Metallurgy, v 28, no 3, 1985
4. YOON, D. N., and HUPPMANN, W. J. *Chemically Driven Growth of Tungsten Grains During Sintering in Liquid Nickel.* Acta. Met., v. 27, 1979, p. 973.
5. LENEL, F. V. *Powder Metallurgy, Principles and Applications.* MPIF, 1980.
6. SHEWMON, P. G. *Transformations in Metals.* McGraw-Hill Book Company, New York, 1969.
7. DIETER, G. E. *Mechanical Metallurgy.* McGraw-Hill Book Company, New York, 1979
8. FLEMINGS, M. C. *Solidification Processing.* McGraw-Hill Book Company, New York, 1974.
9. CARTER, G. F. *Principles of Physical and Chemical Metallurgy.* American Society for Metals, 1979.
10. BARRETT, C. R., NIX, W. D., and TETLEMAN, A. S *The Principles of Engineering Materials.* Prentice-Hall, Inc. New Jersey, 1973
11. YODOGAWA, M. *Effects of Cold Rolling and Annealing on the Mechanical Properties of 90 W-7 Ni-3 Fe Heavy Alloys* Sintering - Theory and Practice, D. Kolar, S. Pejovnik, and M. M. Restic, ed., Elsevier Scientific Publishing Co., 1982.
12. FRANTSEVICH, I. N., TEDOROVICH, O. K., and BAZHENOVA, L. G. *Recrystallization of Tungsten in Tungsten-Nickel-Iron Alloys, Part I.* Soviet Journal of Powder Metallurgy, Metal Ceramics, v 6, 1967, p. 393.
13. NORTHCUTT, W. G., JOHNSON, D. H., FERGUSON, J. E., and SNYDER, W. B. *Variables Affecting the Properties of Tungsten-Nickel-Iron Alloys.* ARPA/ARCOM Kinetic Energy Ammunition Materials Panel and U.S. Army Materials and Mechanics Research Center (AMMRC) Conference, Charlottesville, VA, May 24-27, 1976.
14. WINSLOW, F. R. *The Nickel-Iron-Tungsten Phase Diagram.* Y-12 Plant Report No. 1785, Oak Ridge, Tennessee, 1971.
15. Metals Handbook. 9th ed., v. 9, 1985.
16. PUGH, J. W. *On the Recovery and Recrystallization of Tungsten.* Proceedings of the 3rd Plansee Seminar, F. Benesovsky, Reutte/Tirol, June 1958.
17. PUGH, J. W. *The Temperature Dependence of Preferred Orientation in Rolled Tungsten.* Trans. Met. Soc. of AIME, v. 212, 1958, p. 637
18. KING, G. W., and SELL, H. G. *The Effect of Thoria on the Elevated-Temperature Tensile Properties of Recrystallized High-Purity Tungsten.* Trans. Met. Soc. of AIME, v. 233, 1965, p. 1104.
19. ZUKAS, E. G., and EASH, D. T. *Possible Reinforcement of the Tungsten-Nickel-Iron Composite with Tungsten Fibers.* J. Less Common Metals, v. 32, 1973, p. 345.
20. BEVER, M. B., HOLT, D. L., and TICHNER, A. L. *Progress in Materials Science.* Pergamon Press, Ltd., London, v 17, 1973.
21. HONEYCOMBE, R. W. K. *The Plastic Deformation of Metals.* 2nd ed., American Society for Metals, 1984
22. GERMAN, R. M., and MUNIR, Z. A. *Enhanced Low Temperature Sintering of Tungsten.* Met. Trans A., v. 7A, December 1976, p. 1873.
23. KOTHARI, N. C. *Sintering Kinetics in Tungsten Powder.* J. Less Common Metals, v. 5, 1963, p. 140.
24. VASILOS, T., and SMITH, J. T. *Diffusion Mechanism for Sintering Kinetics.* J. App. Phys., v. 35, no 1, January 1964.
25. KREIDER, K. G., and BRUGGEMAN, G. *Grain Boundary Diffusion in Tungsten.* Trans. Met. Soc. of AIME, v. 239, 1967, p. 1222.
26. GREEN, W. V. *Short Time Creep-Rupture Behavior of Tungsten at 2250°C to 2800°C.* Trans. Met. Soc. of AIME, v 215, 1959, p. 1057.

APPENDIX

Temp (°C)	Time (hr)	Extrusion Ratio	S/V Transverse	S/V Parallel	Extrusion Ratio	S/V Transverse	S/V Parallel
1300	1/2	6:1	0.33	0.04	24:1	0.52	0.07
	1		0.47	0.04		0.50	0.07
	2		0.37	0.06		0.35	0.29
	4		0.20	0.15		0.20	0.18
1350	1/2	6:1	0.35	0.11	24:1	0.47	0.26
	1		0.33	0.19		0.26	0.24
	2		0.20	0.15		0.21	0.18
	4		0.14	0.13		0.25	0.23
1400	1/2	6:1	0.17	0.15	24:1	0.23	0.20
	1		0.20	0.15		0.23	0.15
	2		0.14	0.16		0.20	0.18
	4		0.16	0.14		0.15	0.18
1450	1/2	6:1	0.17	0.18	24:1	0.20	0.20
	1		0.17	0.15		0.20	0.16
	2		0.15	0.11		0.23	0.18
	4		0.13	0.10		0.13	0.14

DISTRIBUTION LIST

No. of Copies	To
1	Office of the Under Secretary of Defense for Research and Engineering, The Pentagon, Washington, DC 20301
	Commander, U.S. Army Laboratory Command, 2800 Powder Mill Road, Adelphi, MD 20783-1145
1	ATTN: AMSLC-IM-TL
	Commander, Defense Technical Information Center, Cameron Station, Building 5, 5010 Duke Street, Alexandria, VA 22304-6145
2	ATTN: DTIC-FDAC
	Metals and Ceramics Information Center, Battelle Columbus Laboratories, 505 King Avenue, Columbus, OH 43201
1	ATTN: Harold Mindlin
	Commander, Army Research Office, P.O. Box 12211, Research Triangle Park, NC 27709-2211
1	ATTN: Information Processing Office
	Commander, U.S. Army Materiel Command (AMC), 5001 Eisenhower Avenue, Alexandria, VA 22333
1	ATTN: AMCLD
	Commander, U.S. Army Materiel Systems Analysis Activity, Aberdeen Proving Ground, MD 21005
1	ATTN: AMXSY-MP, Director
	Commander, U.S. Army Missile Command, Redstone Scientific Information Center, Redstone Arsenal, AL 35898-5241
1	ATTN: AMSMI-RD-CS-R/Doc
1	AMSMI-CS, R. B. Clem
	Commander, U.S. Army Armament, Munitions and Chemical Command, Dover, NJ 07801
2	ATTN: Technical Library
	Commander, U.S. Army Tank-Automotive Command, Warren, MI 48397-5000
1	ATTN: AMSTA-ZSK
2	AMSTA-TSL, Technical Library
1	AMSTA-RCK
	Commander, U.S. Army Foreign Science and Technology Center, 220 7th Street, N.E., Charlottesville, VA 22901
1	ATTN: Military Tech
	Director, Eustis Directorate, U.S. Army Air Mobility Research and Development Laboratory, Fort Eustis, VA 23604-5577
1	ATTN: SAVDL-E-MOS (AVSCOM)
1	SAVDL-EU-TAP
	U.S. Army Aviation Training Library, Fort Rucker, AL 36360
1	ATTN: Building 5906--5907

No. of Copies	To
	Commander, U.S. Army Aviation Systems Command, 4300 Goodfellow Boulevard, St. Louis, MO 63120-1798
1	ATTN: AMSAV-EGX
1	AMSAV-EX, Mr. R. Lewis
1	AMSAV-EQ, Mr. Crawford
2	AMCPM-AAH-TM, Mr. R. Hubbard, Mr. B. J. Baskett
1	AMSAV-DS, Mr. W. McClane
	Naval Research Laboratory, Washington, DC 20375
1	ATTN: Code 5830
1	Code 2627
	Chief of Naval Research, Arlington, VA 22217
1	ATTN: Code 471
	Director, Structural Mechanics Research, Office of Naval Research, 800 North Quincy Street, Arlington, VA 22203
1	ATTN: Dr. M. Perrone
	Commander, U.S. Air Force Wright Aeronautical Laboratories, Wright-Patterson Air Force Base, OH 45433
1	ATTN: AFWAL/MLC
1	AFWAL/MLLP, D. M. Forney, Jr.
1	AFWAL/MLBC, Mr. Stanley Schulman
1	AFWAL/MLXE, A. Olevitch
	National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, AL 35812
1	ATTN: R. J. Schwinghammer, EH01, Dir, M&P Lab
1	Mr. W. A. Wilson, EH41, Bldg. 4612
	Air Force Armament Laboratory, Eglin Air Force Base, FL 32542
1	ATTN: AFATL/DLYA, V. D. Thornton
	Air Force Test and Evaluation Center, Kirtland Air Force Base, NM 87115
1	ATTN: AFTEC-JT
	Naval Post Graduate School, Monterey, CA 93948
1	ATTN: Code 57BP, R. E. Ball
	Naval Surface Weapons Center, Dahlgren Laboratory, Dahlgren, VA 22448
1	ATTN: Code G-54, Mr. J. Hall
1	Code G-54, Dr. B. Smith
	Commander, Rock Island Arsenal, Rock Island, IL 61299-6000
1	ATTN: SMCRI-SEM-T
	Battelle Columbus Laboratories, Battelle Memorial Institute, 505 King Avenue, Columbus, OH 43201
1	ATTN: Mr. Henry Cialone
1	Mr. Robert Fiorentino

No. of Copies	To
	Battelle Pacific Northwest Laboratories, P.O. Box 999, Richland, WA 99352
1	ATTN: Mr. William Gurwell
	GTE Sylvania, Inc., Chemical and Metallurgical Division, Hawes Street, Towanda, PA 18848
1	ATTN: Dr. James Mullendore
	Teledyne Firth Sterling, LaVergne, TN 37086
1	ATTN: Mr. Steven G. Caldwell
1	Mr. Thomas Penrice
	Kennametal, Inc., P.O. Box 231, Latrobe, PA 15601
1	ATTN: Mr. Walter Huckaby
	Los Alamos National Laboratory, P.O. Box 1663, Los Alamos, NM 87545
1	ATTN: Mr. Billy Hogan
	Westinghouse Electric Corp., Advanced Energy Systems, P.O. Box 10864, Pittsburgh, PA 15236
1	ATTN: Mr. William Buckman
	Director, U.S. Army Materials Technology Laboratory, Watertown, MA 02172-0001
2	ATTN: SLCMT-TML
1	Author

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
THE RECRYSTALLIZATION AND RESPHEROIDIZATION OF TUNGSTEN GRAINS IN A TUNGSTEN-HEAVY ALLOY - Robert J. Dowding

AD__________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Key Words

Tungsten alloys
Recrystallization
Heavy metals

Technical Report MTL TR 89-31, April 1989, 17 pp-illus-tables, D/A Project P612105.H84

Refinement of the microstructure of tungsten-heavy alloys, specifically 90 W-7 Ni-3 Fe, has been achieved through heavy cold working followed by a heat treatment that recrystallized and respheroidized the tungsten grains. Hydrostatic extrusion of the alloy to reductions in area of 83% and 96% followed by isothermal annealing gives fine, spherical tungsten grains imbedded within the nickel-iron matrix. The finest grain size achieved was 15 microns for the 96% extrusion when subjected to an anneal at 1300°C for 2 hours. The apparent activation energy for the process was found to be 530 kJ/mole. This value indicates that volume diffusiohn is significant in respheroidizing the grains.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
THE RECRYSTALLIZATION AND RESPHEROIDIZATION OF TUNGSTEN GRAINS IN A TUNGSTEN-HEAVY ALLOY - Robert J. Dowding

AD__________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Key Words

Tungsten alloys
Recrystallization
Heavy metals

Technical Report MTL TR 89-31, April 1989, 17 pp-illus-tables, D/A Project No. P612105.H84

Refinement of the microstructure of tungsten-heavy alloys, specifically 90 W-7 Ni-3 Fe, has been achieved through heavy cold working followed by a heat treatment that recrystallized and respheroidized the tungsten grains. Hydrostatic extrusion of the alloy to reductions in area of 83% and 96% followed by isothermal annealing gives fine, spherical tungsten grains imbedded within the nickel-iron matrix. The finest grain size achieved was 15 microns for the 96% extrusion when subjected to an anneal at 1300°C for 2 hours. The apparent activation energy for the process was found to be 530 kJ/mole. This value indicates that volume diffusiohn is significant in respheroidizing the grains.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
THE RECRYSTALLIZATION AND RESPHEROIDIZATION OF TUNGSTEN GRAINS IN A TUNGSTEN-HEAVY ALLOY - Robert J. Dowding

AD__________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Key Words

Tungsten alloys
Recrystallization
Heavy metals

Technical Report MTL TR 89-31, April 1989, 17 pp-illus-tables, D/A Project No. P612105.H84

Refinement of the microstructure of tungsten-heavy alloys, specifically 90 W-7 Ni-3 Fe, has been achieved through heavy cold working followed by a heat treatment that recrystallized and respheroidized the tungsten grains. Hydrostatic extrusion of the alloy to reductions in area of 83% and 96% followed by isothermal annealing gives fine, spherical tungsten grains imbedded within the nickel-iron matrix. The finest grain size achieved was 15 microns for the 96% extrusion when subjected to an anneal at 1300°C for 2 hours. The apparent activation energy for the process was found to be 530 kJ/mole. This value indicates that volume diffusiohn is significant in respheroidizing the grains.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
THE RECRYSTALLIZATION AND RESPHEROIDIZATION OF TUNGSTEN GRAINS IN A TUNGSTEN-HEAVY ALLOY - Robert J. Dowding

AD__________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Key Words

Tungsten alloys
Recrystallization
Heavy metals

Technical Report MTL TR 89-31, April 1989, 17 pp-illus-tables, D/A Project No. P612105.H84

Refinement of the microstructure of tungsten-heavy alloys, specifically 90 W-7 Ni-3 Fe, has been achieved through heavy cold working followed by a heat treatment that recrystallized and respheroidized the tungsten grains. Hydrostatic extrusion of the alloy to reductions in area of 83% and 96% followed by isothermal annealing gives fine, spherical tungsten grains imbedded within the nickel-iron matrix. The finest grain size achieved was 15 microns for the 96% extrusion when subjected to an anneal at 1300°C for 2 hours. The apparent activation energy for the process was found to be 530 kJ/mole. This value indicates that volume diffusiohn is significant in respheroidizing the grains.